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# CASE FILE

OUTGASSING TESTS ON A MICROWAVE OVEN AND SELECTED FREEZE-DRIED FOODS COOKED IN THE OVEN

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concentration of volatile prod					
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## OUTGASSING TESTS ON A MICROWAVE OVEN AND SELECTED FREEZE-DRIED FOODS COOKED IN THE OVEN

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#### **SUMMARY**

A series of tests was performed at Langley Research Center to determine the outgassing characteristics of a microwave oven and of a variety of freeze-dried foods cooked in the oven in order to determine the environmental effect of preparing hot meals on prolonged space flights. The tests were conducted in a vacuum chamber at a pressure of 68.9 kN/m² (10 psia) to simulate a potential spacecraft atmosphere. The main contaminants from the microwave oven were n-butanol, i-butanol, and hexane. Only one food, chicken chunks, produced a sufficient concentration of volatile products for positive identification. These products were hexanal and methyl formate.

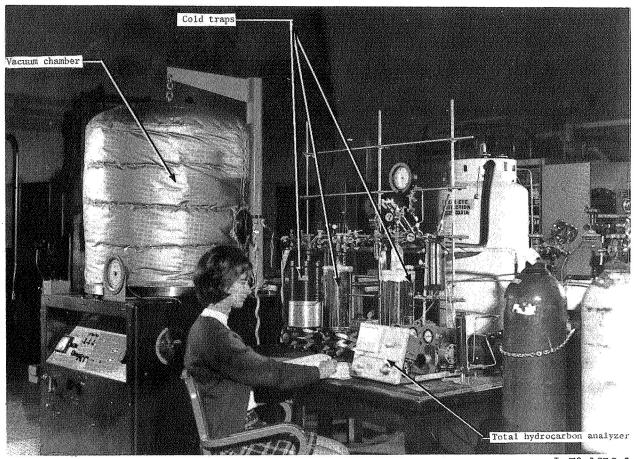
#### INTRODUCTION

As man performs more extensive experiments and explorations in space, the duration of manned missions will of necessity increase. This increase is apparent in the present space program, which has progressed from the early missions which were measured in minutes to later missions measuring in hours and days. At the present time plans for the Skylab program call for missions of 28 and 56 days (ref. 1). Ground-based simulator tests to evaluate life-support equipment and crew performance have been conducted for continuous durations of up to 90 days (ref. 2).

It has long been recognized that food plays an important role with regard to man's performance over a period of time, not only from the viewpoint of nutritional value, but also from that of morale, or psychological well-being (ref. 3). It was primarily from the latter requirement that a decision was made to provide hot meals for the 90-day manned test of reference 2 during which a microwave oven was used to heat reconstituted freezedried foods. Because of the closed environment afforded by the space station simulator (SSS) used for this 90-day test, it was essential to determine, prior to the test, the outgassing contaminants produced by the oven itself and by the various foods to be heated. Previous outgassing tests on foods have been conducted only on fresh and some frozen foods (refs. 4, 5, 6, and 7). Therefore, a series of outgassing tests on freeze-dried foods was conducted in support of the 90-day simulator test. The results of these tests and a description of the test procedures developed are presented in this report.

#### APPARATUS AND TESTS

The major components of the test apparatus are the vacuum chamber, the microwave oven, a set of cold traps, and a total hydrocarbon analyzer. Figure 1 is a photograph of the apparatus.



L-70-1252.1

Figure 1.- Outgas generation and removal apparatus.

#### Vacuum Chamber

The vacuum chamber consists of a vertically oriented metal bell jar which has a free volume of about  $0.5664~\mathrm{m}^3$  and is covered with a heating mantle. The tests were conducted in the vacuum chamber at a pressure of  $68.9~\mathrm{kN/m}^2$  (19 psia) which was the same as the pressure of the SSS during the 90-day manned test. Room air was used as the chamber atmosphere. The oxygen level in the vacuum chamber was approximately 20 percent by volume while the simulator oxygen level was approximately 30 percent by volume.

#### Microwave Oven

The oven is identical to the one selected for the 90-day test; however, electrical circuits were modified for the present tests so that the cooking cycle could be activated remotely from outside the vacuum chamber. The oven was mounted on supports in the fixed-base portion of the vacuum chamber (see fig. 2).



L-70-1250

Figure 2.- Microwave oven situated in base of vacuum chamber.

#### Food

The freeze-dried foods used in these tests were obtained from the same manufacturer as the foods used in the 90-day test. A list of the foods tested in this experiment and their weights is presented in table I.

TABLE	I	FOOD

Food	Weight,	Food	Weight,
Crab imperial	75	Chunk chicken with rice and carrots*	80
Pork chops	45	Dried eggs	38
Beef tenderloin	75	Pork and potatoes au gratin	83
Fish chowder*	60	Ham and green beans au gratin	82
Turkey tetrazzini	64	Chopped broccoli	7
Chicken chop suey*	70	Chicken chunks	50

 $<sup>^*</sup>$ These foods were used in both the outgassed products analysis and the outgassing rate measurements.

These foods were representative of the meats, seafood, vegetables, and combinations of meats and vegetables that were consumed by the crew during the 90-day test. Each portion of food tested was equal to the quantity normally consumed by one man at one meal. Cooking time was approximately 45 seconds.

#### Cold Traps

Samples of the outgassed products were obtained from a series of cold traps, as shown in the schematic drawing in figure 3. These traps were mounted in series with the inlet and return lines attached to the outgassing site. The three traps were immersed in ice water, a mixture of dry ice and Dowanol, and liquid nitrogen. The liquid-nitrogen trap was operated at one-half atmosphere to reduce the condensation of oxygen. Flow rate through the trapping system was maintained between 45 and 50 cm<sup>3</sup>/min. Several hours of trapping were required to freeze out sufficient quantity of the outgassed products for identification.

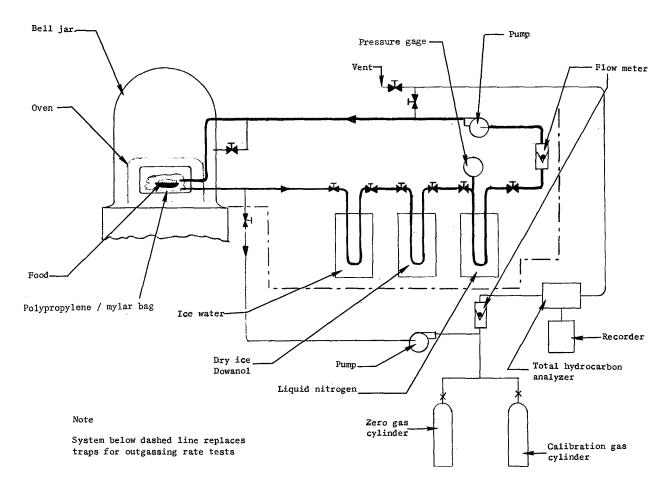


Figure 3.- Schematic drawing of test systems.

The ice-water and dry-ice and Dowanol traps were all-glass systems which were cleaned and rinsed with distilled water between uses. The liquid-nitrogen trap was stainless steel and was cleaned by pulling approximately 1-mm vacuum while heating to 473 K for 12 hours.

#### Test Procedure

Outgassing-products tests were made of the vacuum chamber alone, the oven installed in the chamber, and the freeze-dried foods. In each of these tests, the chamber pressure was reduced to  $68.9~\rm kN/m^2$  (10 psia) and the trapping was maintained for several hours.

In the test of the chamber alone no outgassing products were detected. On the test of the oven alone several small peaks were measured on the gas chromatograph. In order to concentrate these small peaks, tests were run with the oven, without food, encased in a polypropylene-mylar laminated bag which was attached directly to the trapping lines. During the pump down, the pressure in the bag was equalized with the chamber pressure by an externally operated valve. Once the desired pressure, 68.9 kN/m² (10 psia), was achieved, the valve was closed. The oven was first tested cold and then heated to 366.5 K using the heating mantle surrounding the vacuum-chamber bell jar.

Because of the oven outgassing all food tests were made by sealing the food in small polypropylene-mylar bags which were fastened to the trapping lines. The food was first put into a paper dish, the correct amount of boiling water added, and the food stirred. The dish was then immediately put into the bag, the bag put into the oven, and the lines attached (see fig. 3). The oven timer was set and the chamber closed and pumped down. The bag was vented to the chamber during pump down and when 68.9 kN/m² (10 psia) was reached the valve was closed so that only the volatiles from the food were trapped. After pump down the cooking cycle was initiated and the trapping process begun.

Two series of tests were run on the food servings — the first to determine the outgassing products and the second to determine the outgassing rate.

At the completion of each trapping operation the traps were sealed and removed from the system. The traps were heated in a water bath to volatilize the trapped products. Each of the trap outlets was fitted with a septum and, by the use of gas-tight syringes, samples were withdrawn and injected into the gas chromatograph. Since some of the traps were at a reduced pressure, a two-way valve was installed between the sampling syringe and the syringe needle for accurate measurement of sample volume.

All traps were analyzed. The ice-water trap contained large amounts of water and no other products could be identified. All the qualitative and quantitative data were determined from the dry-ice and Dowanol and the liquid-nitrogen traps.

Known amounts of each outgassed compound were injected into the gas chromatograph and, by the measurement of peak heights, calibration curves were drawn. The calibration curves were checked each day. The samples were measured by comparing their peak heights with the calibration curves.

#### Instrumentation

The outgassing rates were estimated from time histories of the total volatiles measured by a total hydrocarbon analyzer that was installed in the trapping lines in place of the cold traps. The hydrocarbon analyzer is equipped with a diaphragm pump to maintain a flow rate of approximately  $10 \text{ cm}^3/\text{min}$ . The rate tests provided a measure of the contaminant level that might be expected during actual preparation of the food. The hydrocarbon analyzer is accurate to  $\pm 1$  part per million.

A Varian gas chromatograph, equipped with a  $1.8-m \times 0.05$ -cm i.d. Porapak Q column using a helium ionization detector and run isothermally at 384 K, was set up for the measurement of sulfides, mercaptans, and acrolein. Detection of these compounds was emphasized since they were considered to be hazardous to the crew in low concentrations. The lower limit of detection of these compounds is 10 ppm.

The major portion of the analysis was run on an F & M 810 gas chromatograph with a  $6.1\text{-m} \times 0.05\text{-cm}$  i.d. 20 percent Carbowax 20M on Chromosorb W column using a flame ionization detector. The instrument was run in two modes, isothermally at 375 K and programed from 298 to 375 K. A post column stream splitter was used with this chromatograph so that the eluting compound could be trapped for injection into the mass spectrometer.

The eluting compounds were collected in 10.16-cm 22-gage stainless-steel syringe needles which were equipped with teflon seated two-way valves and were immersed in liquid nitrogen. After collecting a component of interest the trapping system valve was closed and the needle removed from the liquid nitrogen. The sample was immediately exhausted into a Time-of-Flight Mass Spectrometer.

#### RESULTS AND DISCUSSION

The results of the outgassing tests are summarized in table II. Only one food, chicken chunks, produced a sufficient concentration of volatile products for positive identification. A chromatogram of the chicken chunks, figure 4, indicates the presence of hexanal (peak no. 2) and methyl formate (peak no. 1). Hexanal and methyl formate have been previously identified as volatiles of fresh food (refs. 4, 5, and 6).

TABLE II.- OUTGASSED PRODUCTS (FOOD AND OVEN)

Peak Product	Mode of identificationa	Detection limit, ppm	Total amount, <sup>b</sup> mg			
			Oven (in bag)		Food (in bag)	
			Hot	Cold	roou (in bag)	
1	Methyl formate	MS	1			0.5 (chicken chunks)
2	Hexanal	MS	1			2.0 (chicken chunks)
3	n-hexane	GC, MS	1	33.0	0.25	
4	2-butanone	GC, MS	1	1.8	.09	
5	Nonane	GC, MS	1	7.5	.22	
6	1, 1, 2-trichlorethylene	GC, MS	1	4.6	.22	
7	Toluene	GC, MS	1	1.5	.10	
8	i-butanol	GC, MS	1	21.6	.27	
9	n-butanol	GC, MS	1	13.2	.27	
10	Isomer of a C <sub>6</sub> olefinic hydrocarbon	MS	1	18		
11	Acetone	GC	1	.7		
12	Ethanol	GC	1	1.3		
13	Ethylbenzene	GC, MS	11	6.3		

<sup>&</sup>lt;sup>a</sup> MS = Mass spectrometer

 $<sup>^{\</sup>mbox{\scriptsize b}}$  Sum of the results of the dry-ice and Dowanol and liquid-nitrogen traps.

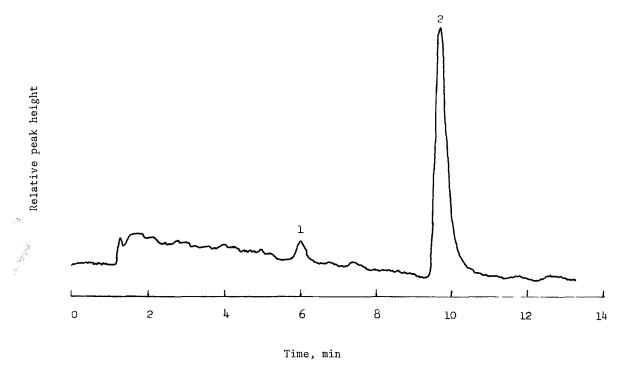


Figure 4.- Chromatogram of chicken chunks in polypropylene-mylar bag. (Sample from dry-ice and Dowanol trap analyzed isothermally at 375 K.)

GC = Gas chromatograph

The sulfides and mercaptans previously reported in tests of fresh foods (refs. 4, 5, 7, 8, and 9) did not appear in sufficient quantities (greater than 10 ppm) to be detected in the present tests.

Chromatograms of the outgassed products from the cold and hot oven tests are presented in figures 5 and 6, respectively. The compounds eluted from the oven are believed to be those generally associated with solvents used in processing the oven materials and during the fabrication and assembly of the oven components. It should be pointed out that the oven used in the test is a commercial product and would not necessarily be fabricated from the same material for a space application.

The possibility of operating the oven in an over-heated condition is unlikely because of built-in safeguards which limit the application of heat only to the vicinity of the cooked foods. The hot oven tests do point up the problems that would exist if the oven were in the vicinity of an external source of heat during the 90-day manned test.

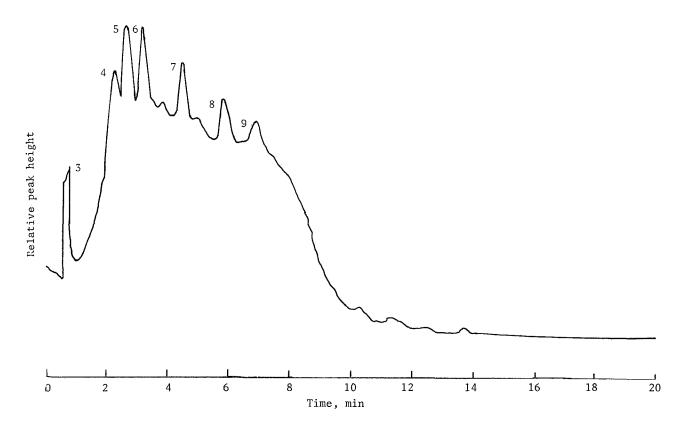


Figure 5.- Chromatogram of cold oven. (Sample from dry-ice and Dowanol trap analyzed isothermally at 375 K.)

The quantities of outgassing products measured are significant for the 90-day test only when related to the outgassing rate and the test termination level established for the 90-day test. The outgassing-rate tests indicate that approximately 75 percent of the volatiles are given off within 2 to 4 minutes after the food is heated in the oven. A slight amount of outgassing could also be anticipated when the food is reconstituted with hot water.

The contaminants obtained in the trapping operation were collected over an 8-hour period of time in order to accumulate enough of the outgassed products to identify by means of the analytical equipment. These values were used, together with the outgassing rate measurements, to calculate the approximate level of contaminants which could be generated in the SSS during the 90-day test period. For example, 2 mg of hexanal (an aldehyde having a test termination level of 25 ppm) were trapped from chicken. Since it was determined from the rate data that about 75 percent of the total outgassing occurs during the normal oven operating period, it could be assumed that about 1.5 mg of hexanal

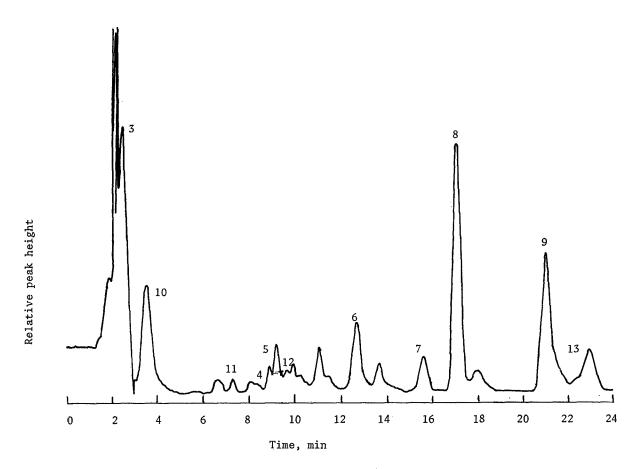


Figure 6.- Chromatogram of oven heated to 366.5 K. (Sample from dry-ice and Dowanol trap programed from 298 to 375 K.)

will outgas for each portion per man per meal. In the worst case, if chicken were eaten at each meal by four crewmen for the entire 90 days, approximately 1620 mg of hexanal would accumulate in the cabin atmosphere, provided no removal was available. The total concentration in the SSS (116  $\rm m^3$ ) would amount to approximately 14  $\rm mg/m^3$  which is equivalent to 4.9 ppm in the simulator.

From the empty cold oven 0.27 mg of n-butanol was measured over an 8-hour time span. Assuming this to be generated at a constant rate with no removal available over a 90-day period, approximately 72.9 mg would accumulate in the cabin atmosphere. The total concentration in the SSS would amount to 0.628 mg/m³ which is equivalent to 0.30 ppm. The test termination level established for the 90-day test was 150 ppm. In the worst case, assuming an oven temperature of 366.5 K for 90 days, the concentration of n-butanol would amount to approximately 30.7 mg/m³ which is equivalent to 14.80 ppm. Similar calculations were done for all other products detected and they were found to be within acceptable limits.

#### CONCLUDING REMARKS

It is evident from the tests performed in order to determine the environmental effect of preparing hot meals on prolonged space flights that the products outgassed from selected portions of freeze-dried food should not exceed the contaminant limits during a 90-day test in a  $116-m^3$  space station simulator even without contaminant removal. It is also shown that the outgassed products from the microwave oven itself would not exceed the established test termination level during the 90-day test.

Langley Research Center,
National Aeronautics and Space Administration,
Hampton, Va., August 30, 1971.

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